In-Situ Real Time Measurement of Melt Constituents

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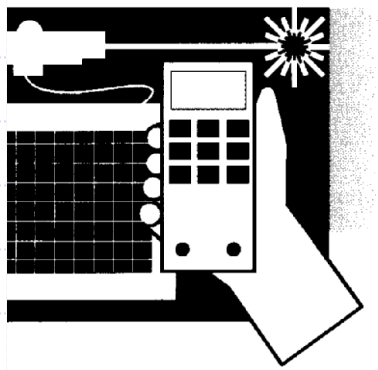
Agenda

- Participants
- Brief Summary of Accomplishments
- Problem Statement
- Concept Description
- Experimental Data
- Software Developments

DOE Participants

- DOE's Office of Industrial Technologies
  - Sensors and Controls
  - Innovation and Inventions
  - Glass



Funding Partners

- DOE
- ERCo
- Crucible Specialty Metals
- NYSERDA
- NY State DED

Participants

- Oak Ridge National Laboratory
- Fenton
- Crucible Specialty Metals
- Alcoa
- Hydro Aluminum
- Crestwood Metals
- Mississippi State University
- University of South Carolina
- Stein Atkinson Stordy

Accomplishments

- Demonstrated Technique
- Inexpensive Probe Developed – Any angle, and depth.
- Probe has 130 hours with No Degradation
- Accuracy Targets Achieved

Accomplishments

- Lab Testing Completed
- Pilot-Scale Testing Underway and Nearly Completed
- Alloy Diffusion Studies Started
- Developing Proprietary Signal Enhancement Techniques

Accomplishments

- ERCo Laser Facility Built
- Proprietary Software Running
  - First Principles
  - No Calibration Needed
  - Excellent Results

Accomplishments Continued

- Will Display at Alcoa/DOE Showcase in August
- Host Sites
  - Alcoa
  - Hydro Aluminum
  - Others

Accomplishments

- For Aluminum, Commercialization Plan Completed
- MOU for Licensing Agreement Signed for Overseas and US Market
- Patent in Process

Accomplishments

- Other Opportunities
  - Glass Batch – DOE Funding
  - Molten Steel – NYSERDA Funding
  - Alloy Identification – NY DED Funding

Problem Statement

- Off-Line Sampling of Melt Constituents on a Batch, Intermittent Basis
- This Leads to
  - Excessive Melting Times
  - Quality Problems
  - Wasted Feedstock
  - Increased Energy Use and Emissions
  - Wasted Product

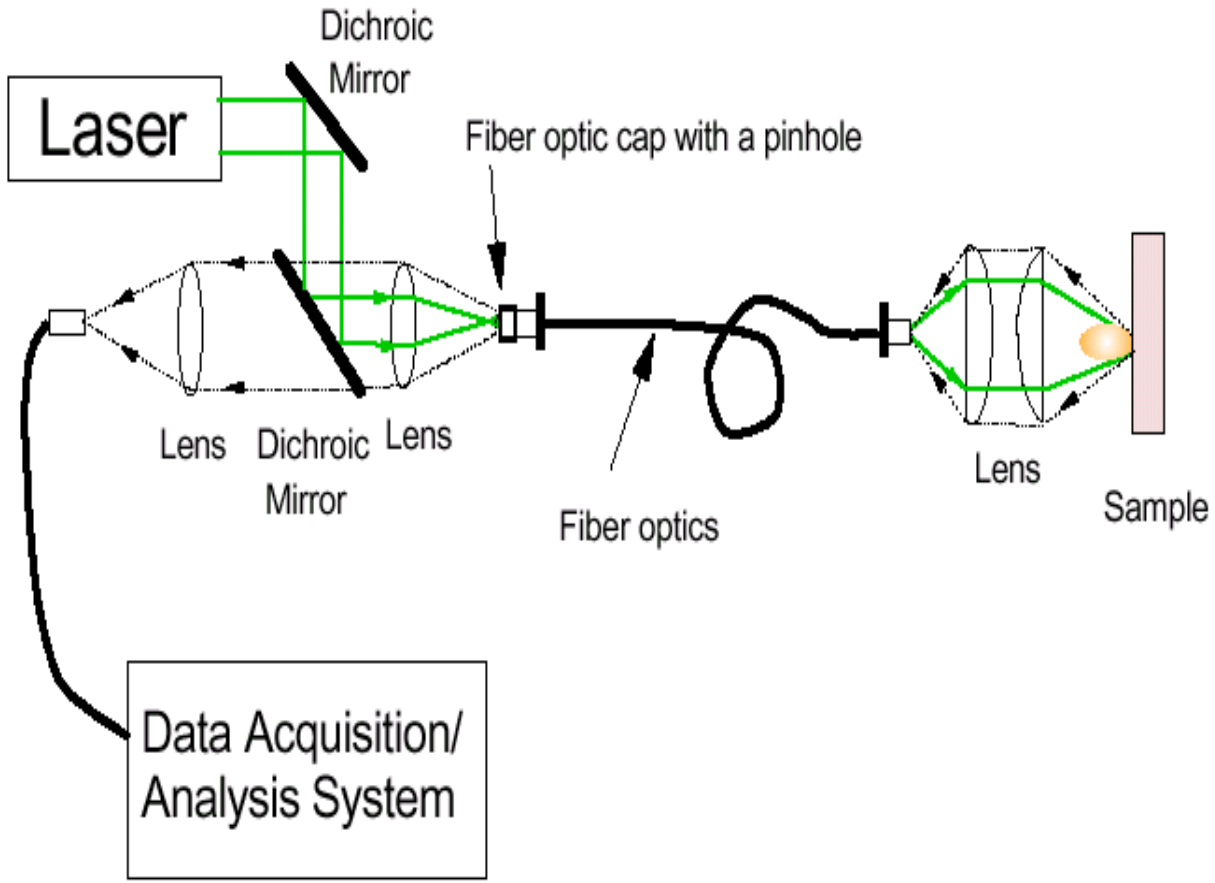
Goals

- Identify elemental constituents in metal and glass melts during alloying and fabrication process
- Develop an instrument capable of:
  1. Being inserted into the melt
  2. Taking real-time, continuous measurements
  3. Installed costs acceptable to industry

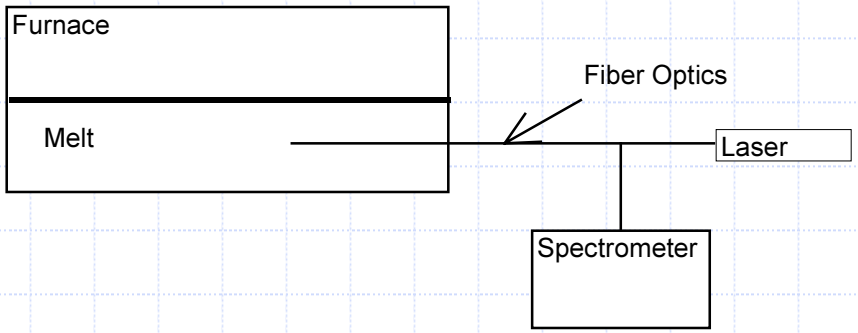
LIBS Technology

- Laser Induced Breakdown Spectroscopy
- Tightly focused laser is used to vaporize a minute amount of material resulting in a plasma
- UV light emitted by the plasma is analyzed using a spectrometer
- The strength of emissions from individual elements in the spectrum are directly related to their concentration in the material

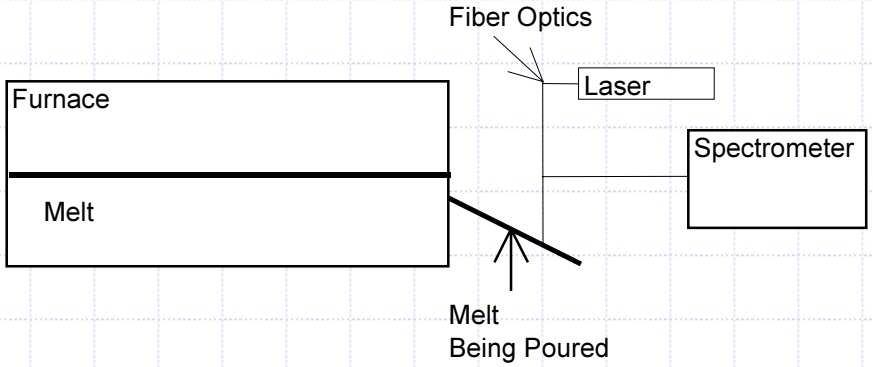
LIBS Optical Design



Concept Description



A. Measurements Made Within Furnace

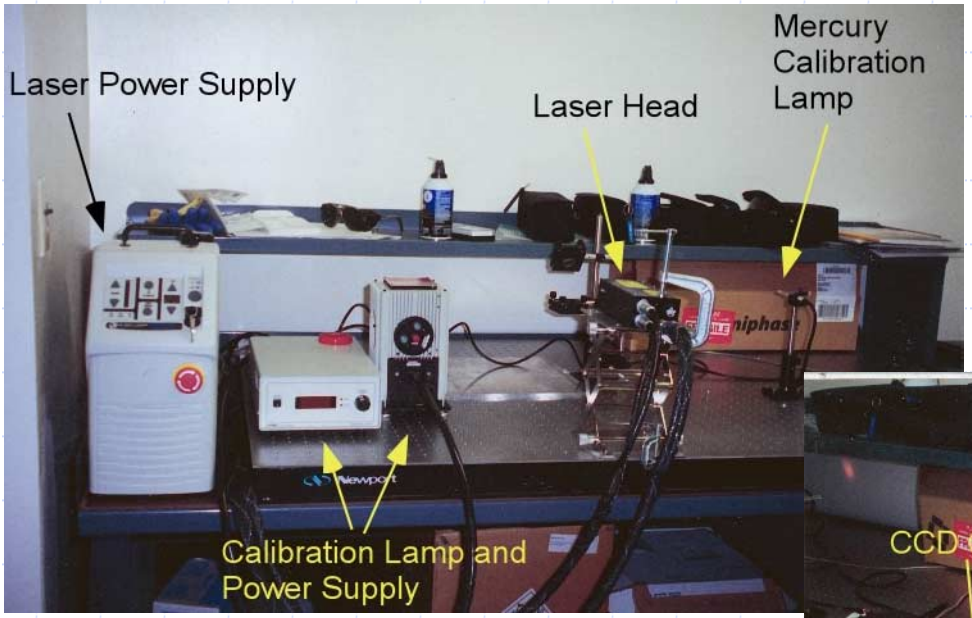


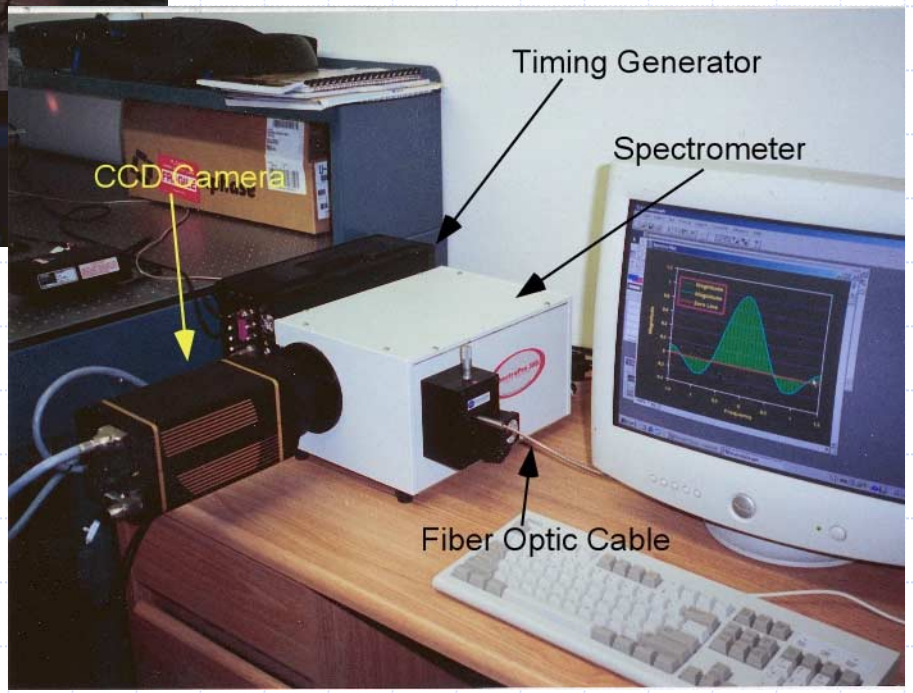
B. Measurements Made During Pour

Aluminum Applications

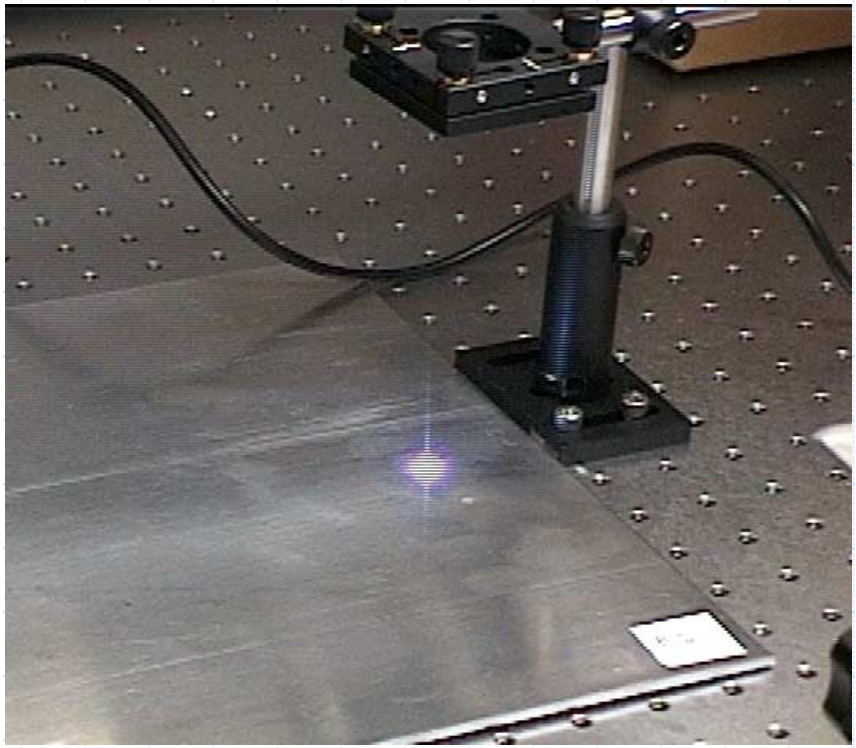
- Aluminum - In-Line Alloying
- Aluminum - Continuous Furnace
- Advance Science of Furnace Modeling
- Aluminum Scrap Alloy Separation

ERCo LIBS Facility



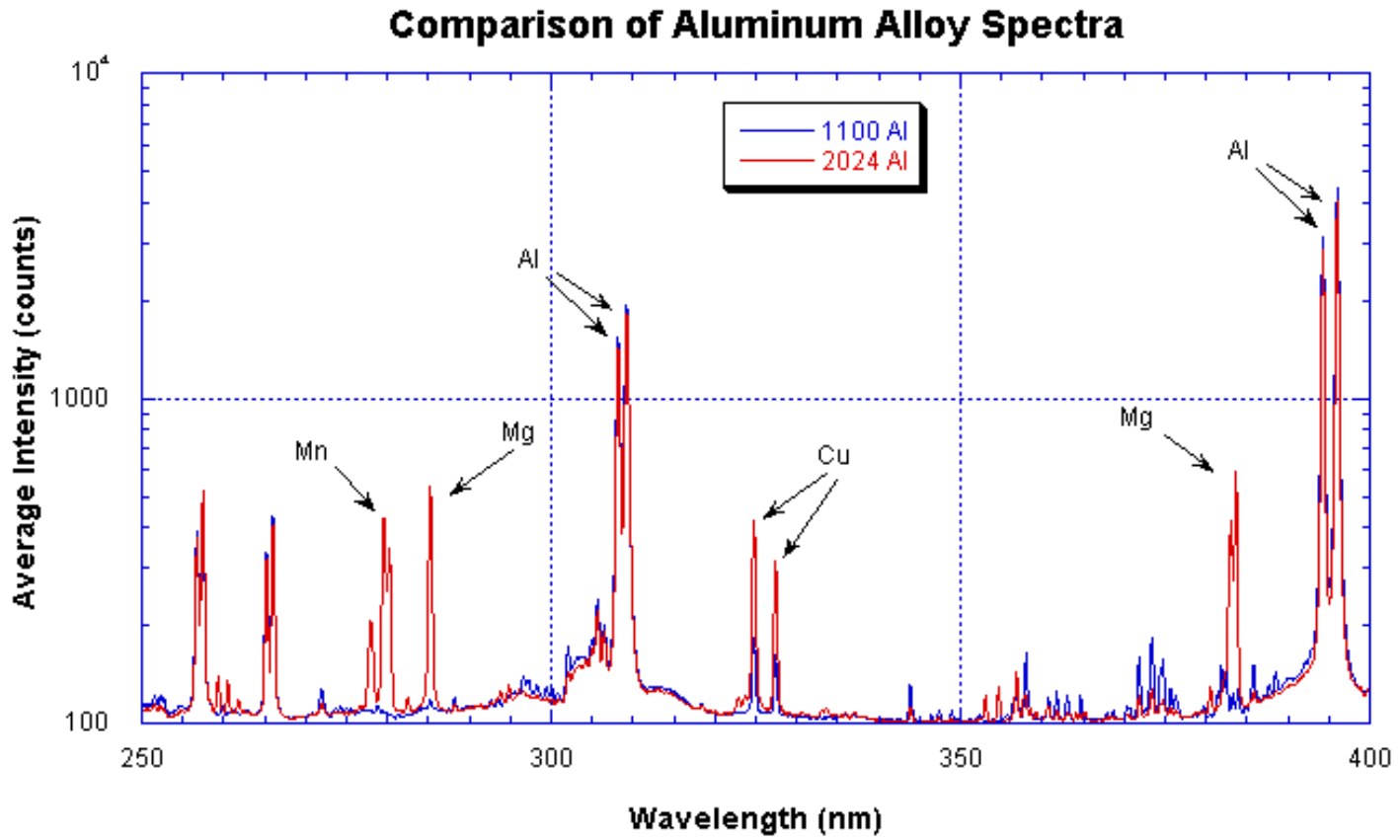


LIBS Technology



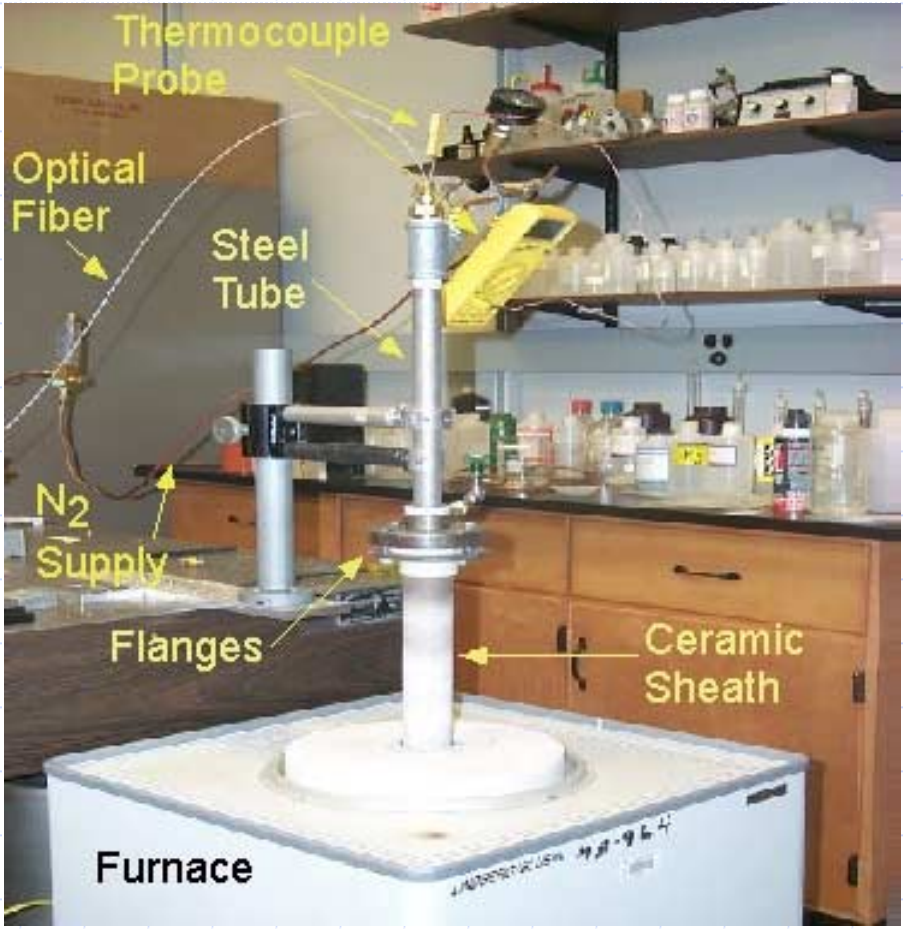
A Tightly Focused Pulsed Laser Is Used to Vaporize and Ionize the Sample Material (Solid, Liquid, or Gas)

LIBS Technology

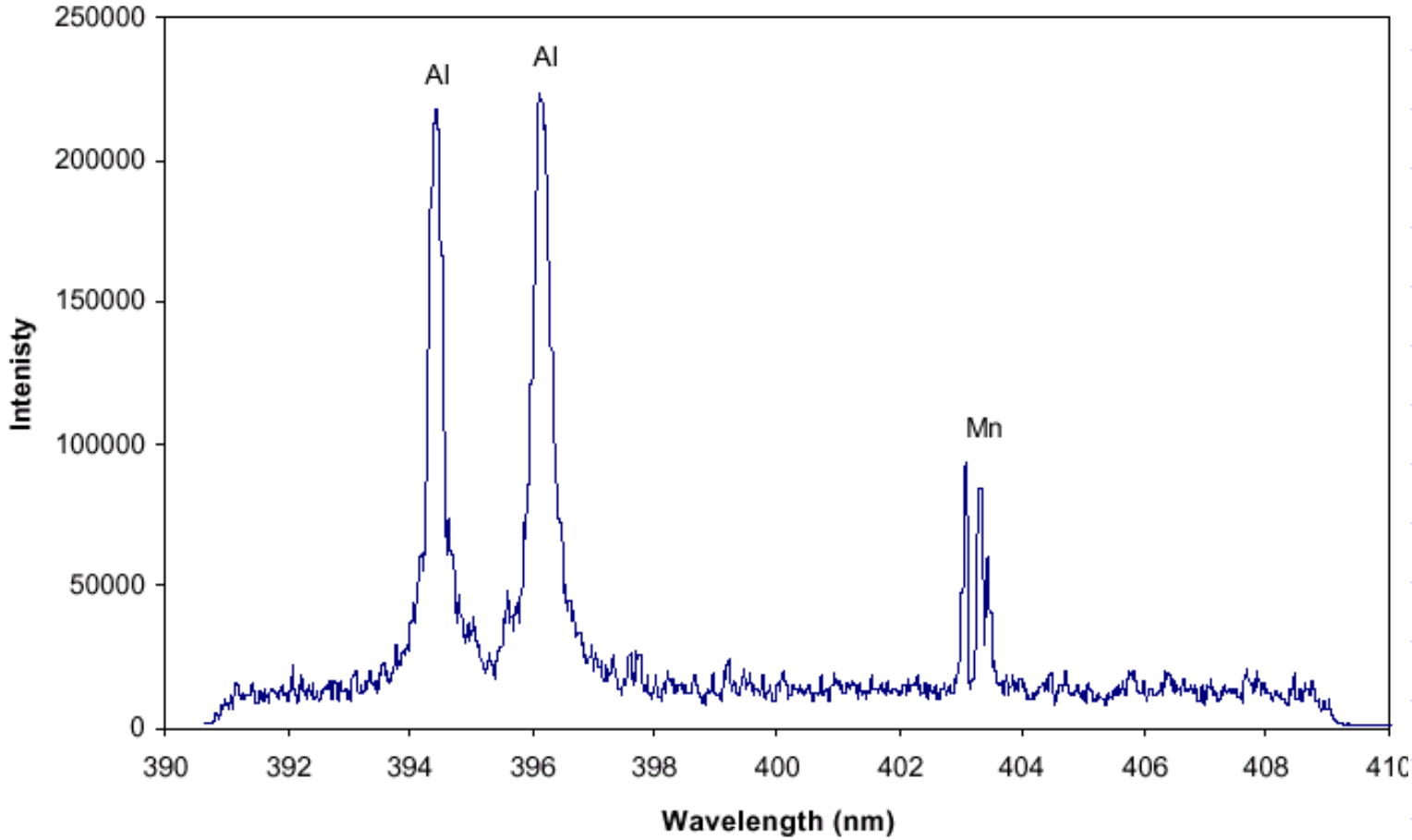


Spectrometer gathers the ultraviolet light and spreads it, like a prism, into a spectrum where the contribution of each element can be seen

Laboratory Scale Probe



LIBS Data – Molten Aluminum



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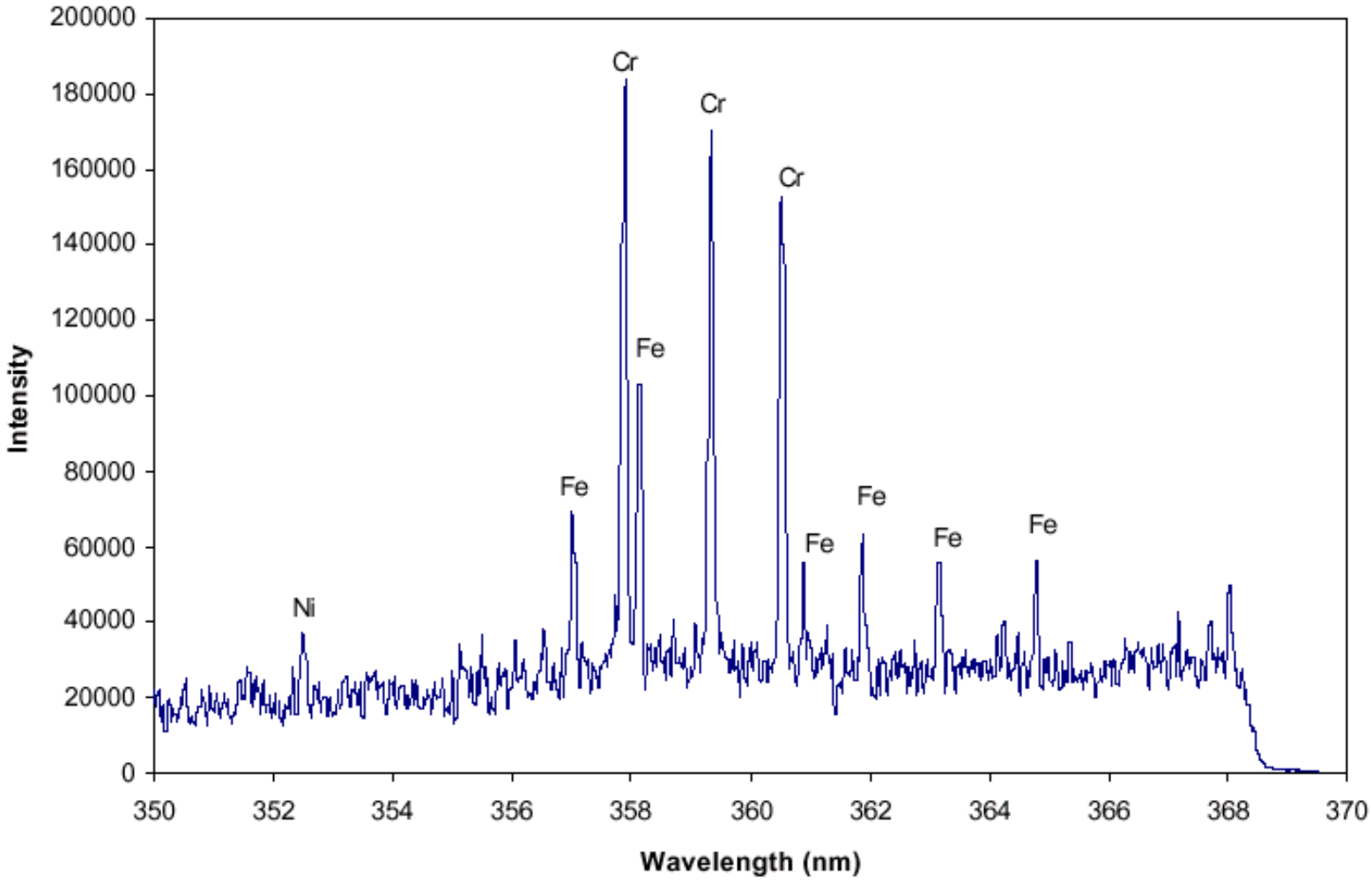
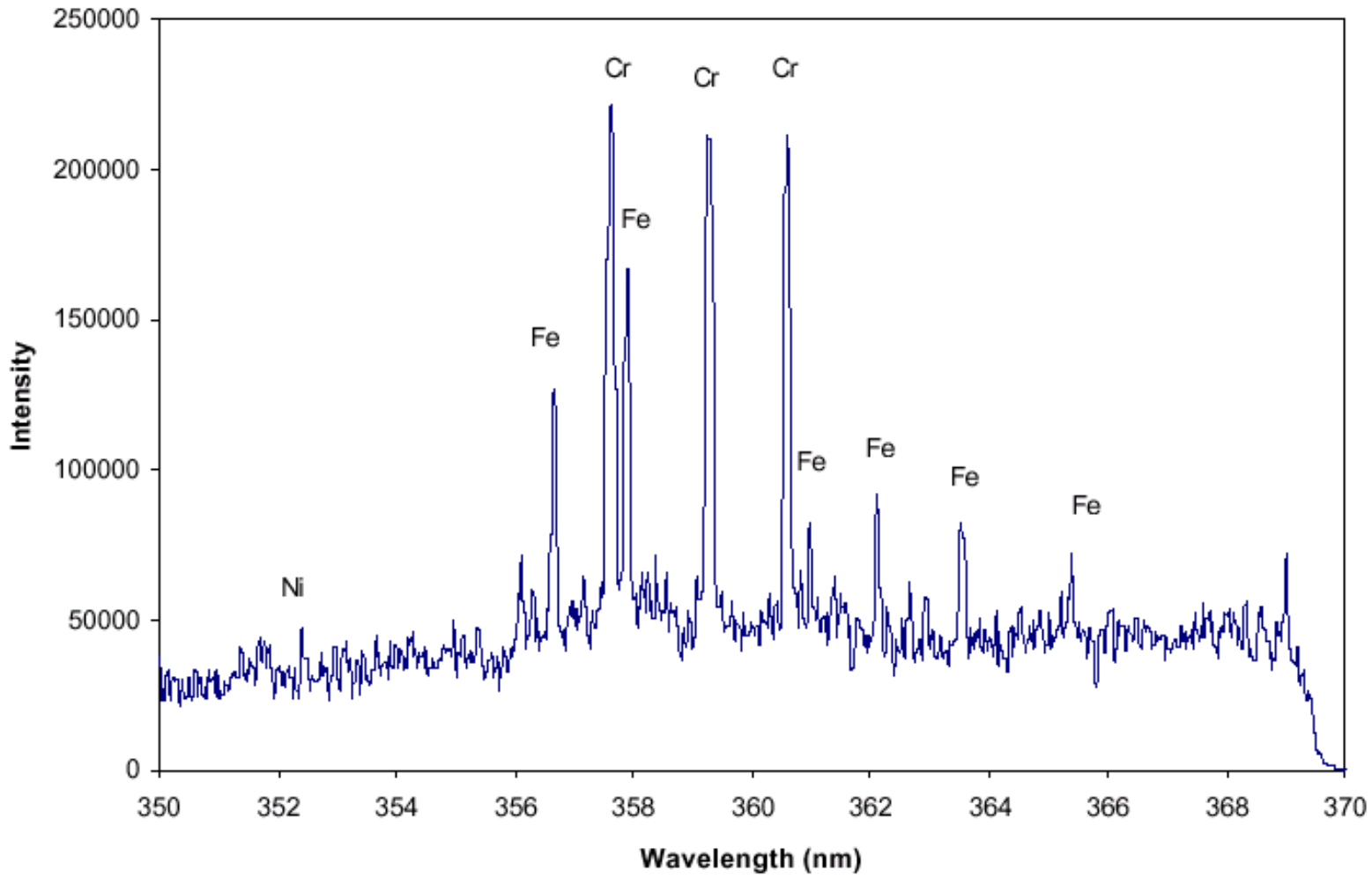


LIBS Data From Solid Plate

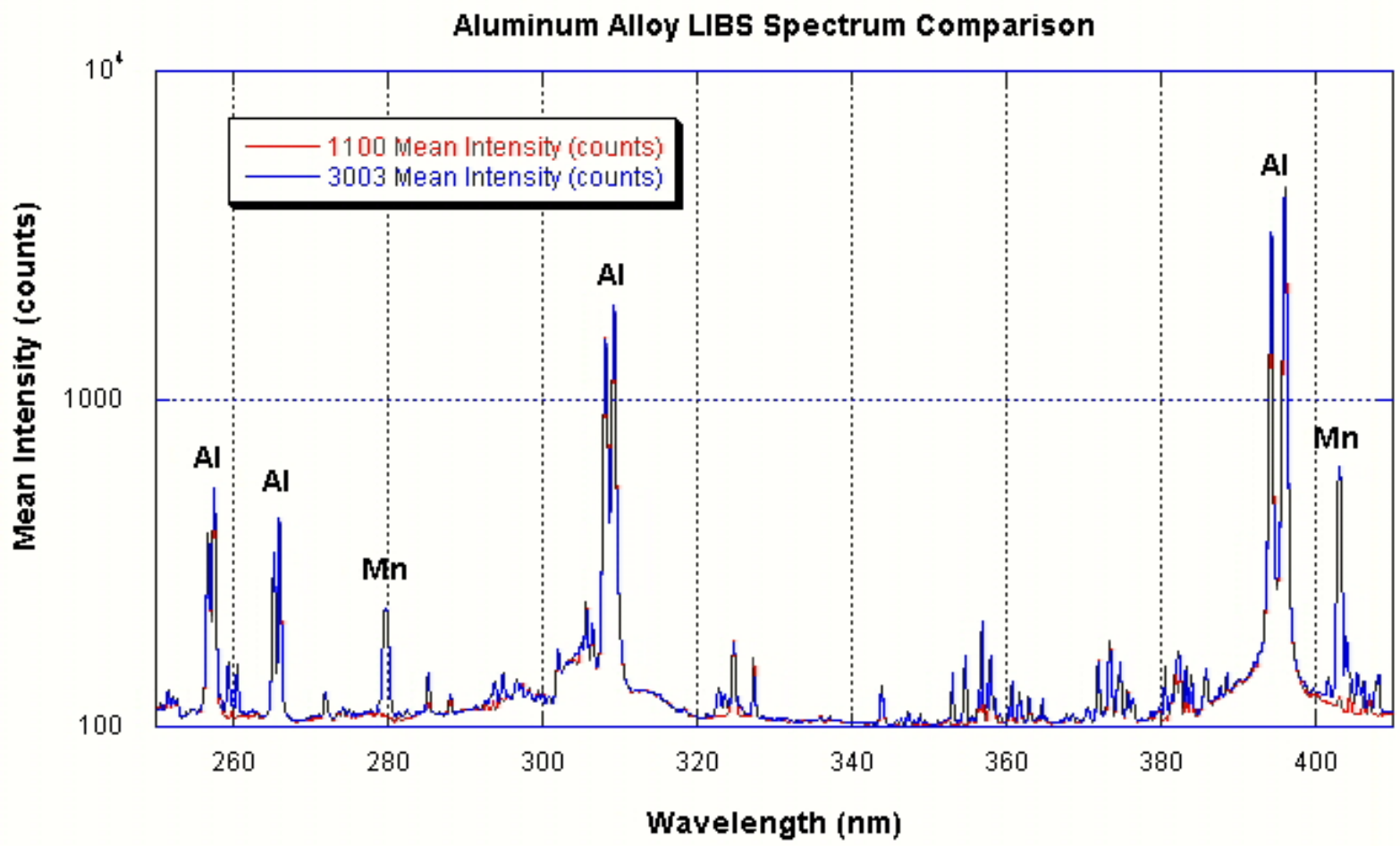


Cr Measurements

Spectral Peaks From Mean Spectrum of Inside Molten 6061 Aluminum

	Peak Signal (1000's counts)	Local Noise Level (1000's counts)	Peak-Noise (1000's counts)	Difference From Plate
Cr 357.9 nm	182	34	148	0.68 %
Cr 359.3 nm	170	34	136	-3.55 %
Cr 360.5 nm	153	34	119	-1.65 %

Data Analysis



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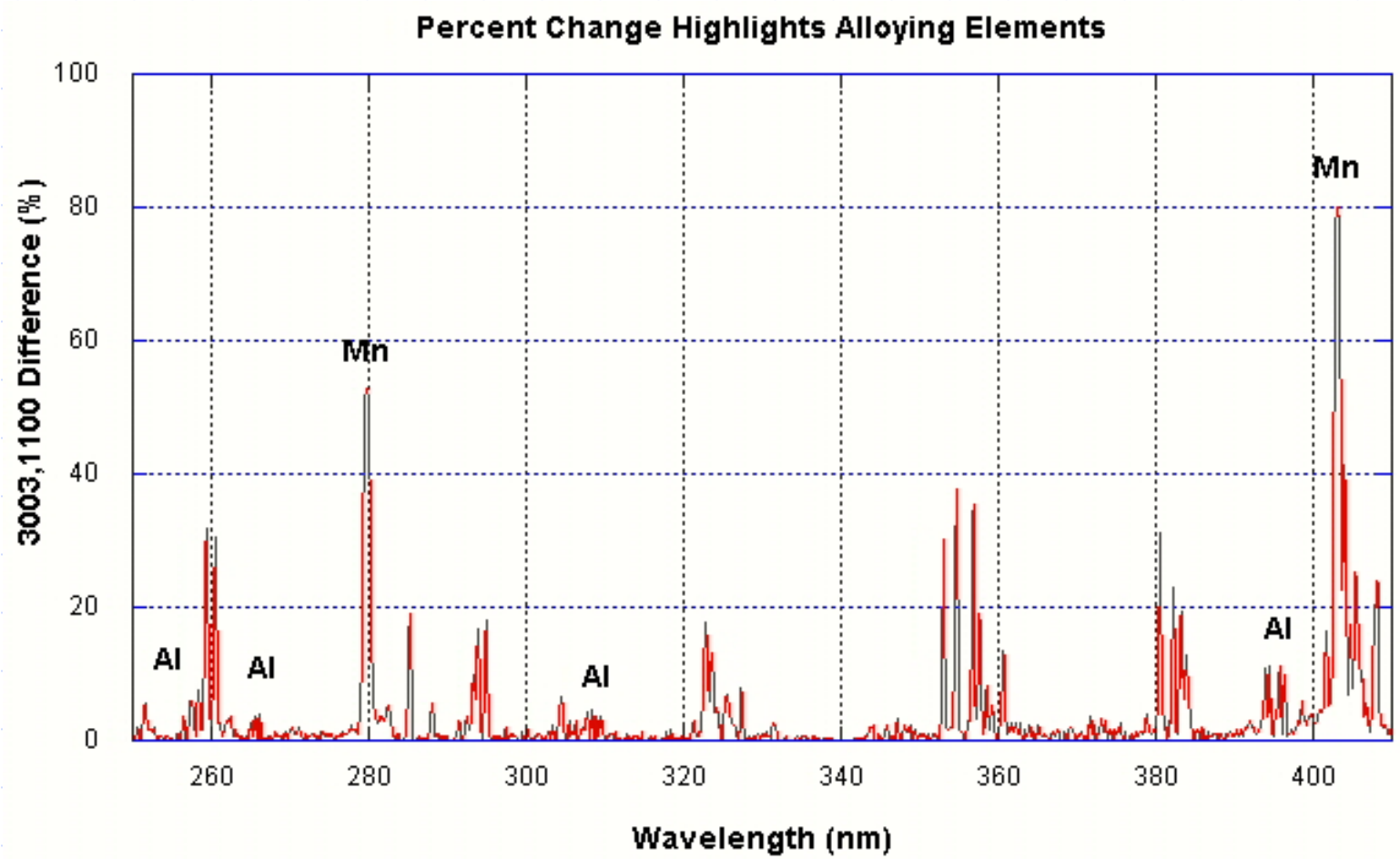
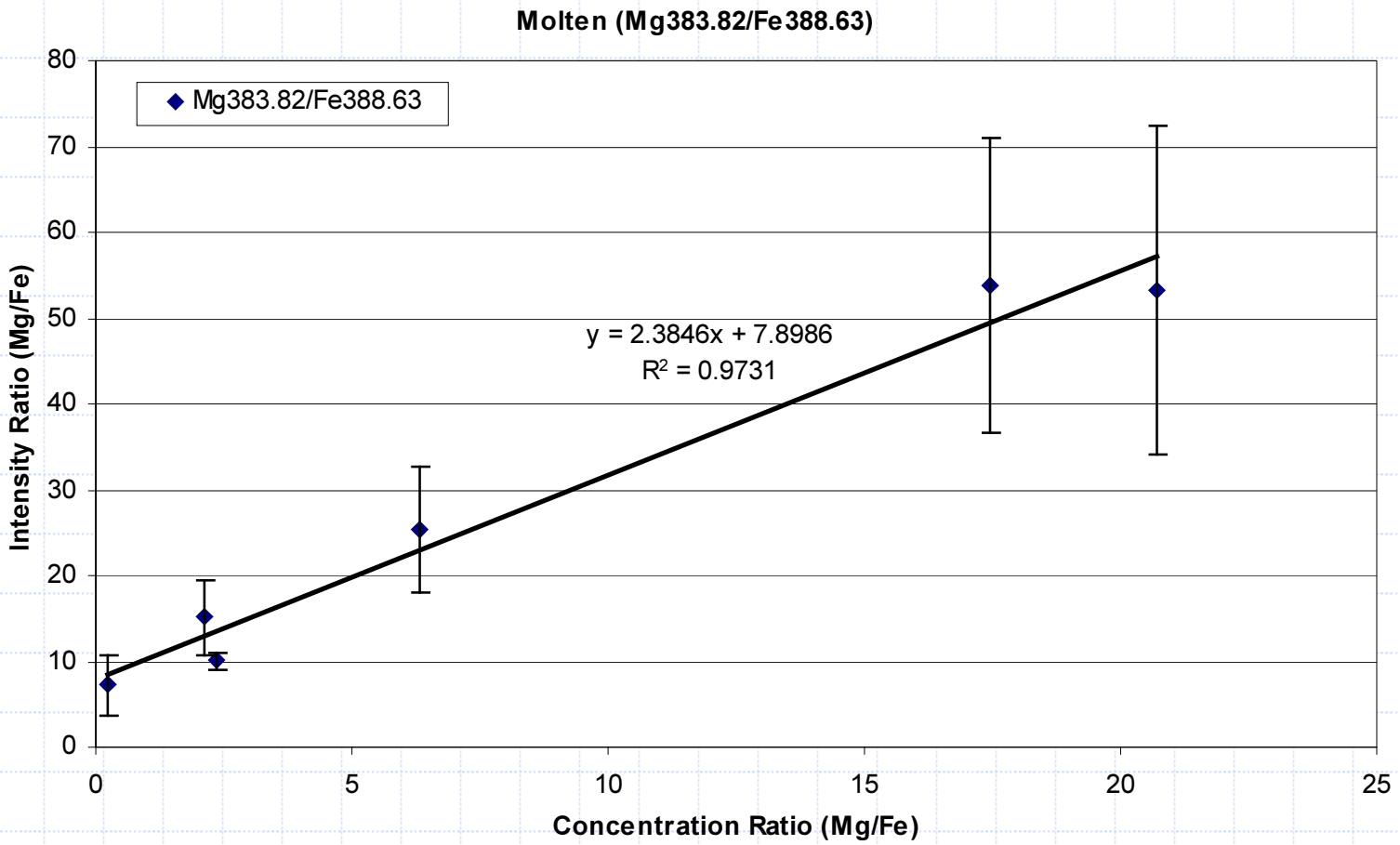


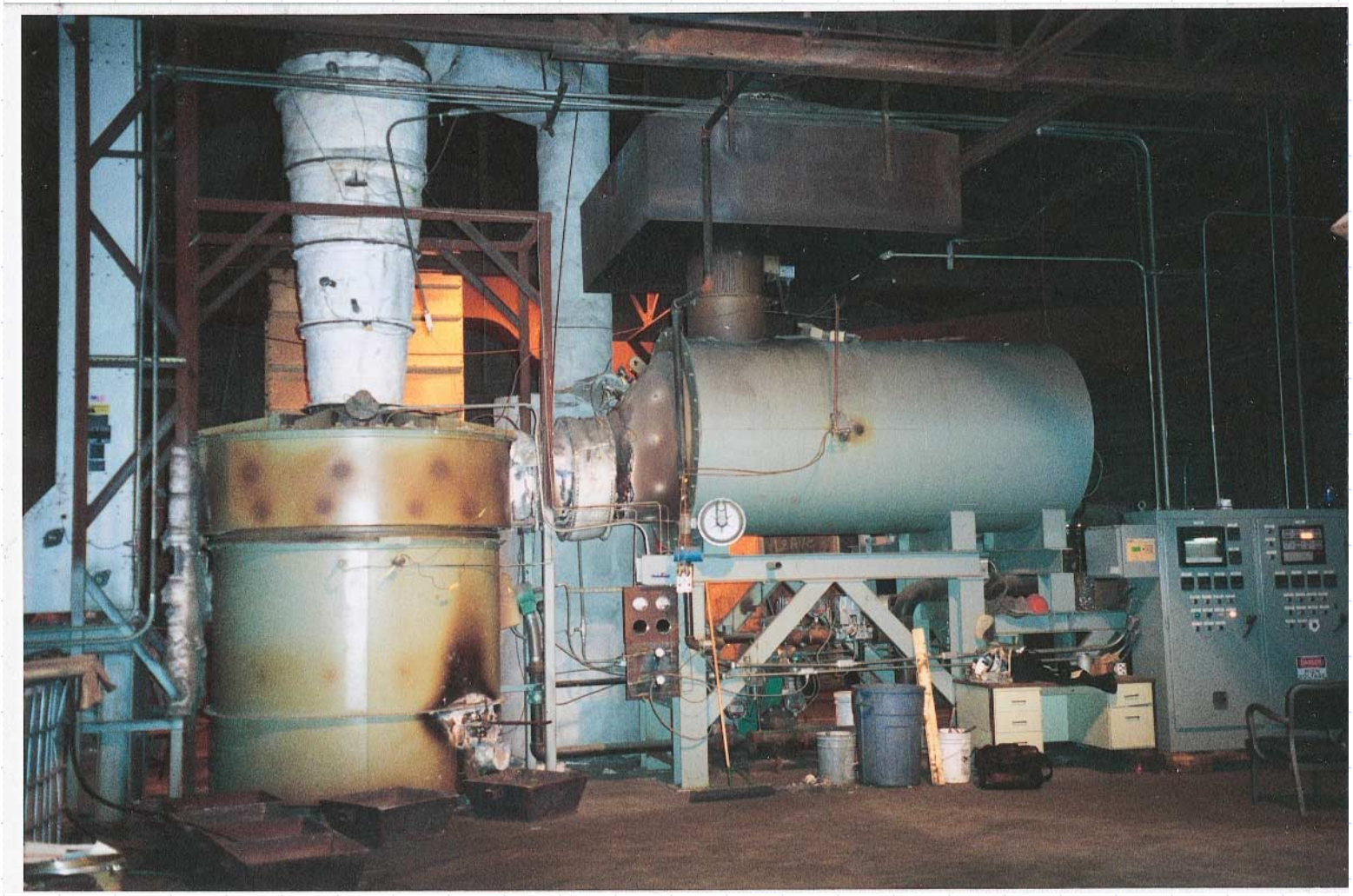
Calibration Curves



Durability

- Probe was immersed in molten aluminum for a total of 130 hours at 800 ºC (1470 ºF)
- Probe is still operable:Neither probe sheath nor the optics suffered degradation

VFM



Installation at Pilot Scale Facility

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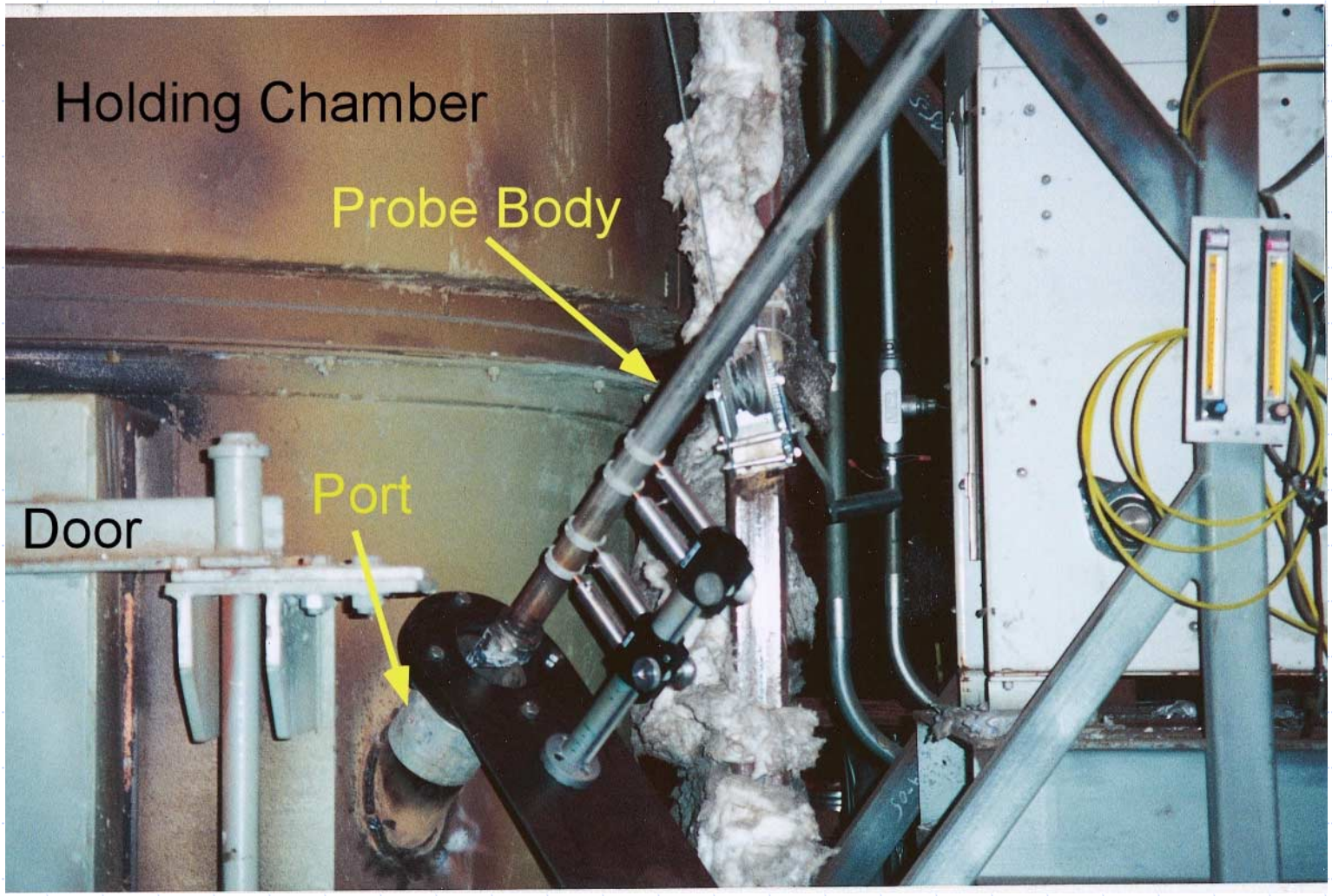

Pilot Scale Testing

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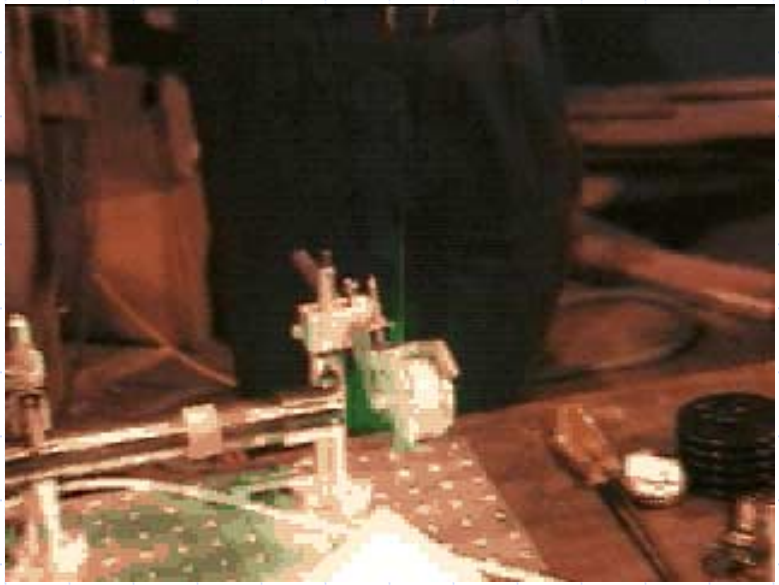
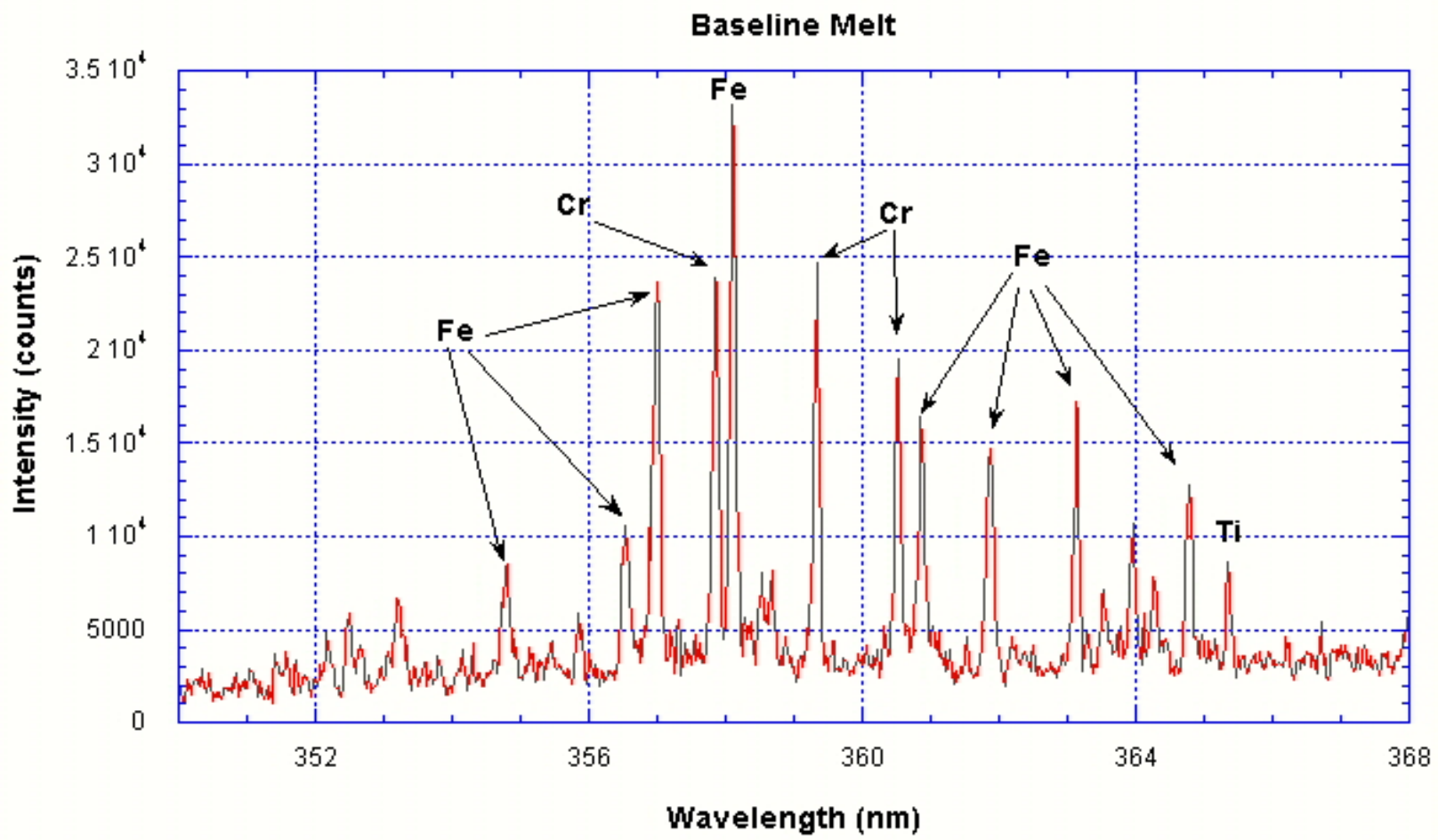


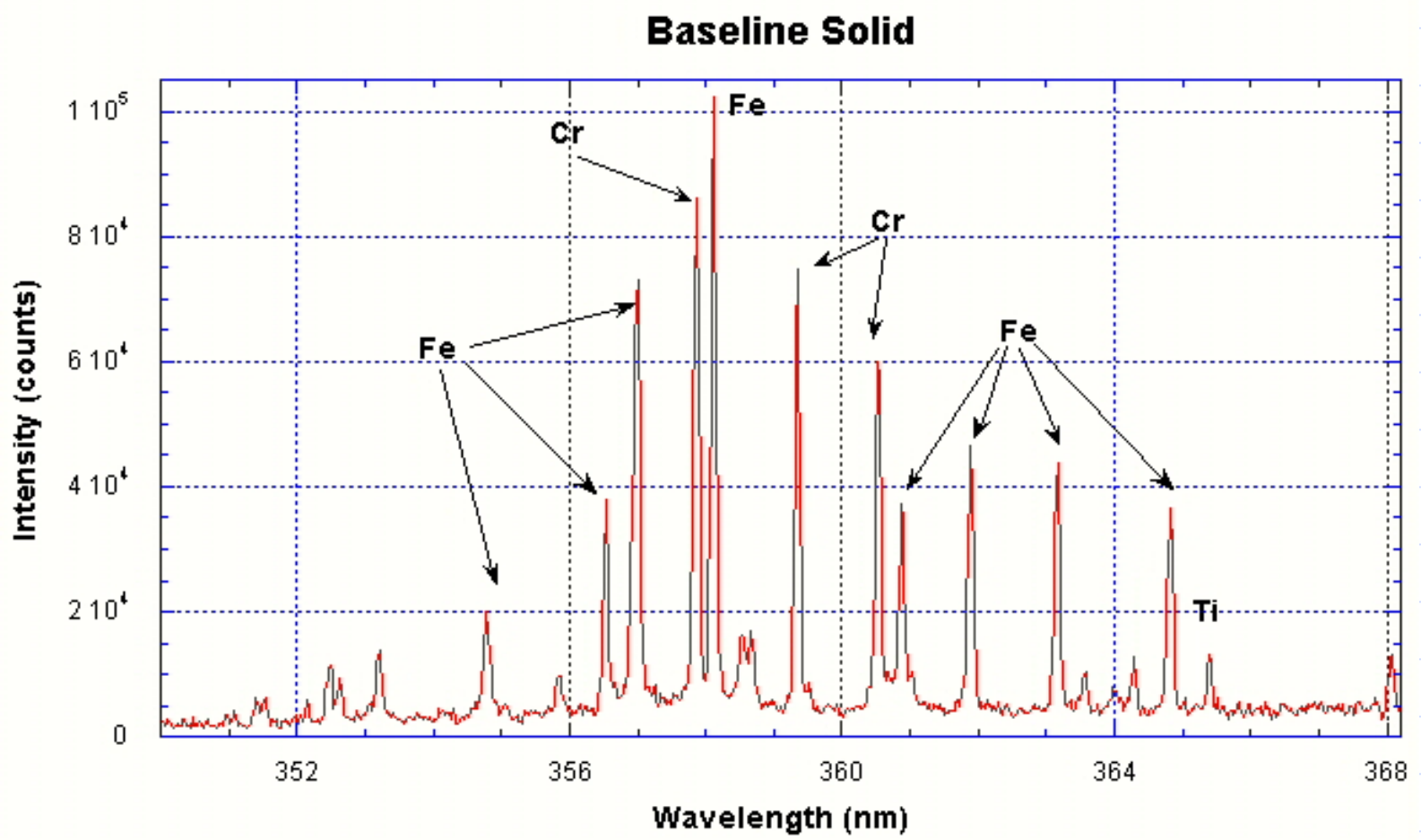
Pilot Scale Test Plan

1. Measure molten base metal formulation and compare to LIBS spectra from solid sample
2. Add Magnesium to change concentration by 0.1% and compare to base metal spectra
3. Add Magnesium, Copper, Chromium, and Manganese to change concentration by 0.2-0.3% and compare to base metal spectra

LIBS Data – Aluminum



LIBS Data - Aluminum



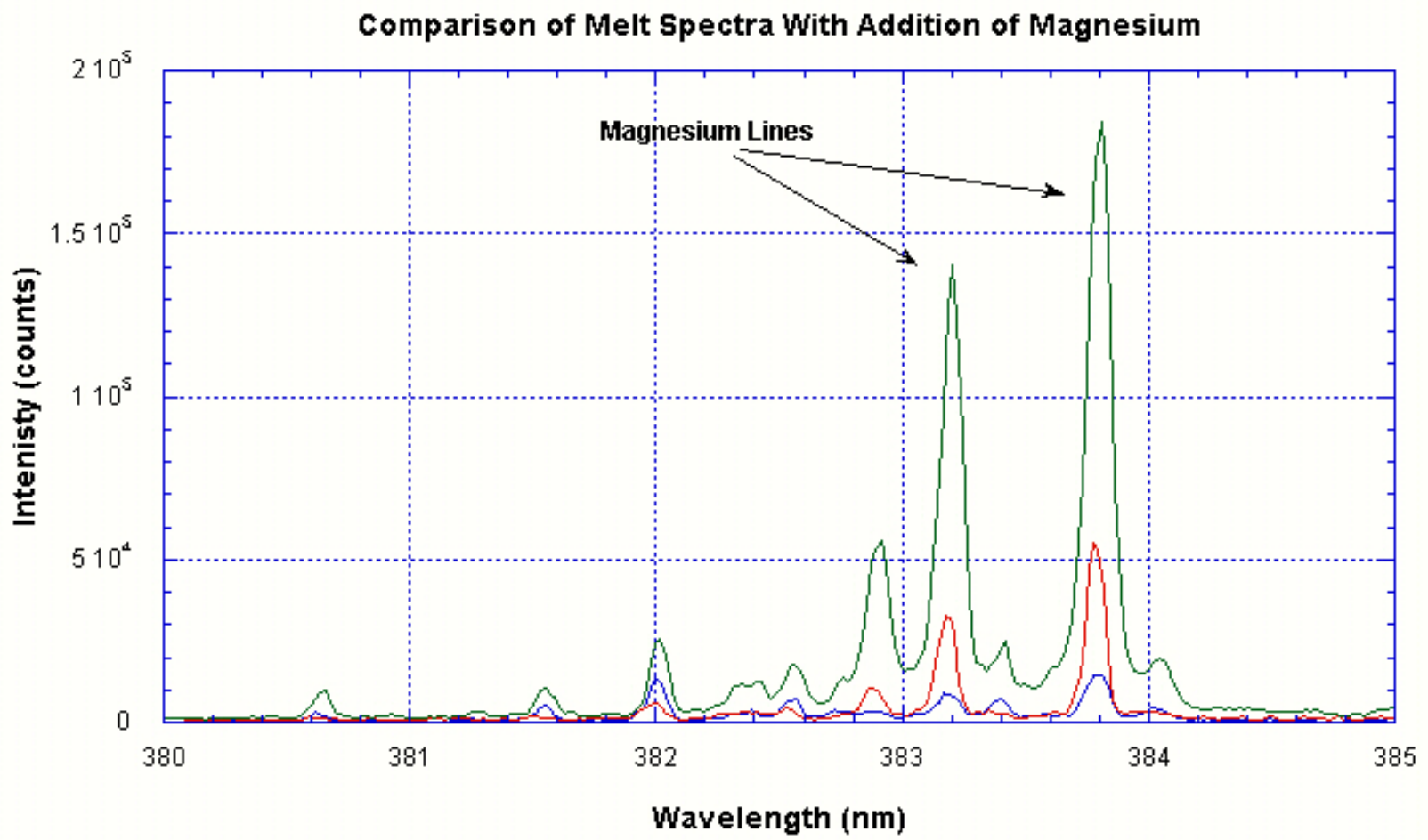
Concentration Measurements

- Calculate Ratios of Elements from Line Intensity Ratios

Element Ratio	Baseline Ratio	Measured Line Ratio	Calibrated Ratio
Fe/Al	0.0078	0.56[1]	0.0079
Mn/Fe	0.51	1.00[2]	0.504
Cr/Al	0.0003	0.48[2]	0.0003

1=Molten, 2=Solid

LIBS Data – Change in Composition

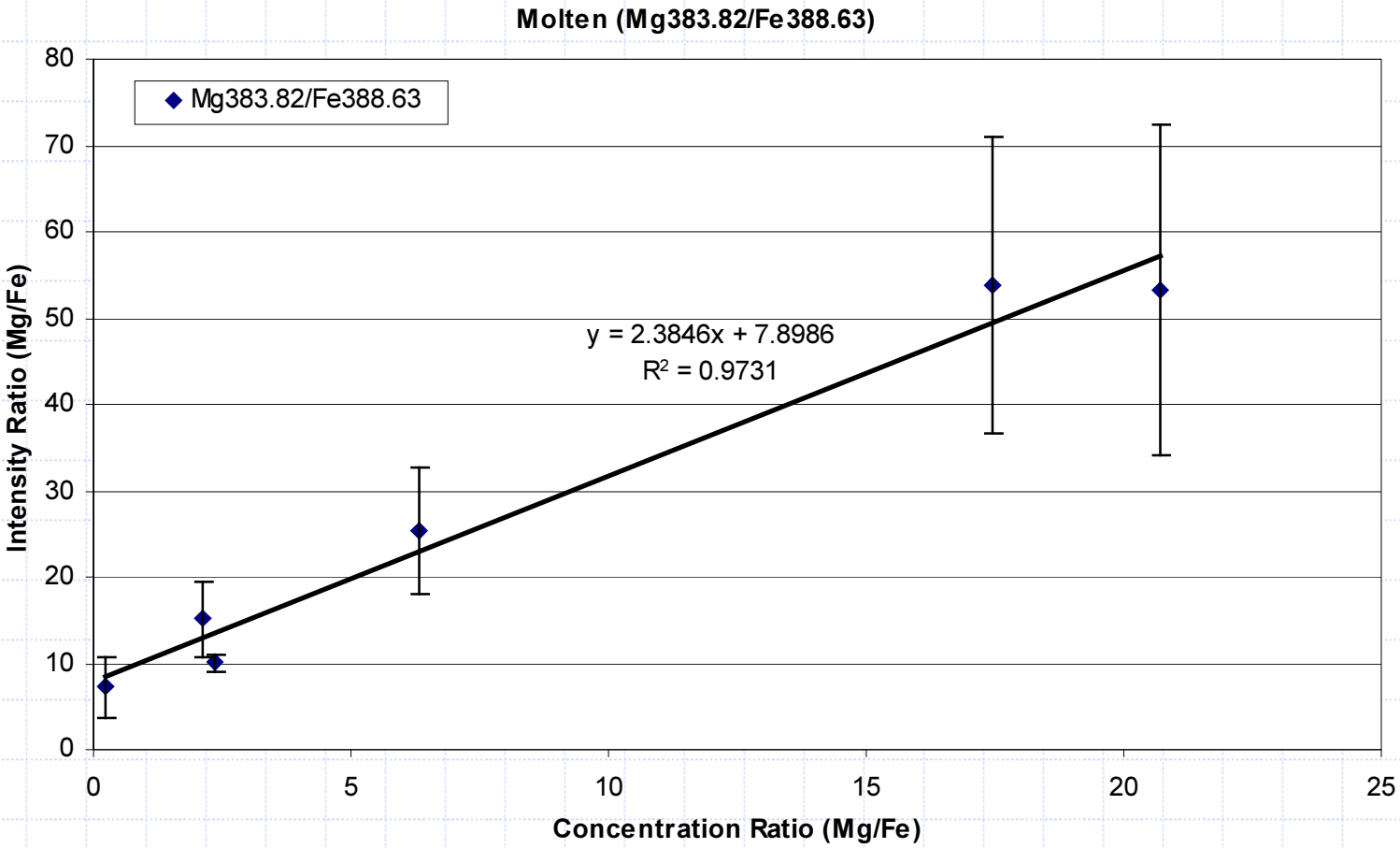


Concentration Measurements

Element Ratio	Added Percentage	Calibrated Ratio Increase
Mn/Fe	0.2%	0.21%[1]
Mg/Fe	0.2%	0.32%[1]

1=Molten, 2=Solid

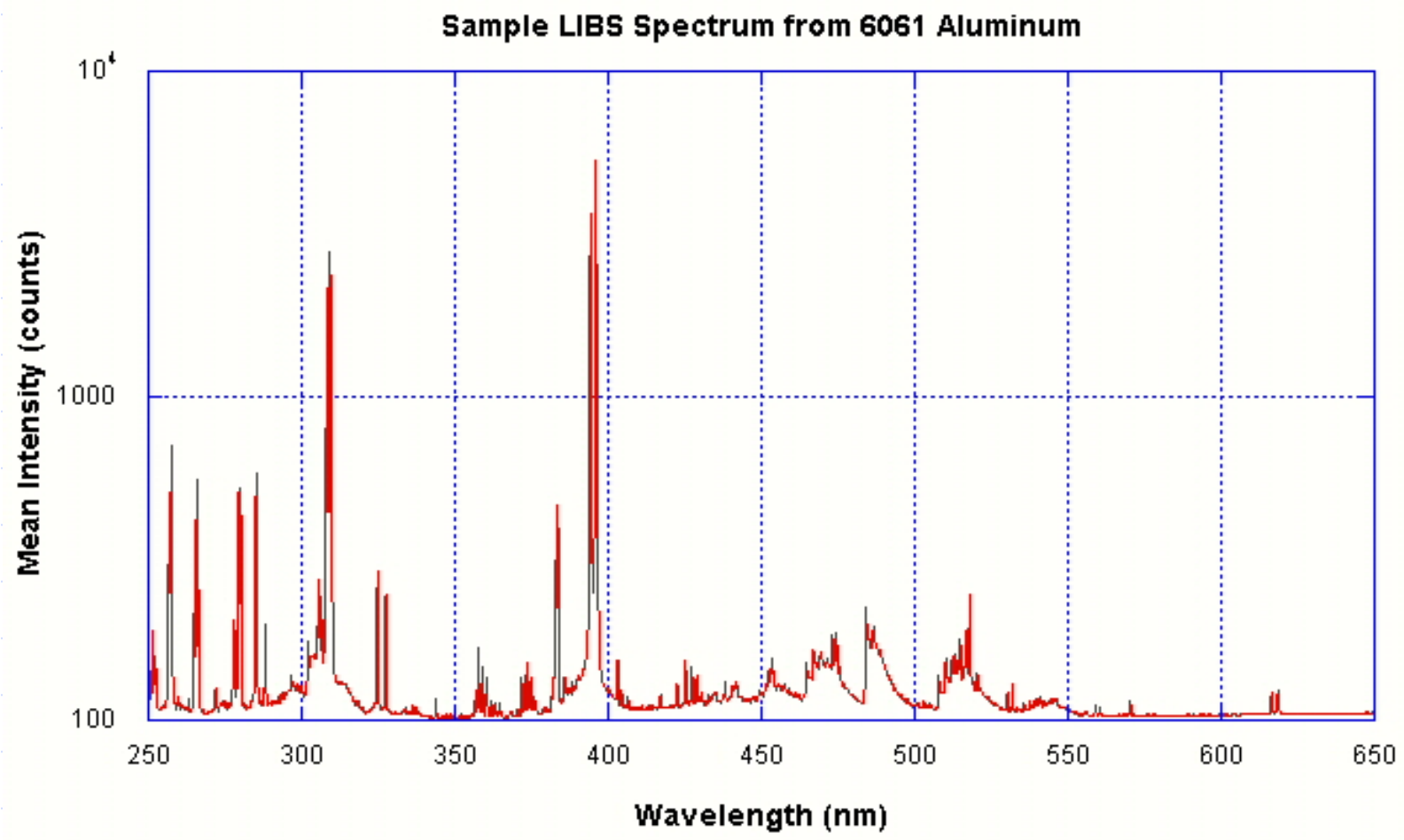
Calibration Curves



Software Developments

- ERCo has Proprietary Software Using First Principles that Translates LIBS Spectra into Concentration Measurements
- Method Applies to both Molten and Solid Data
- Method is Independent of Experimental Parameters such as Laser Power
- No Calibration Data Required
- Actual Concentrations are Computed (rather than ratios)
- Sample Material Does Not Need to be Specified

Initial Software Test



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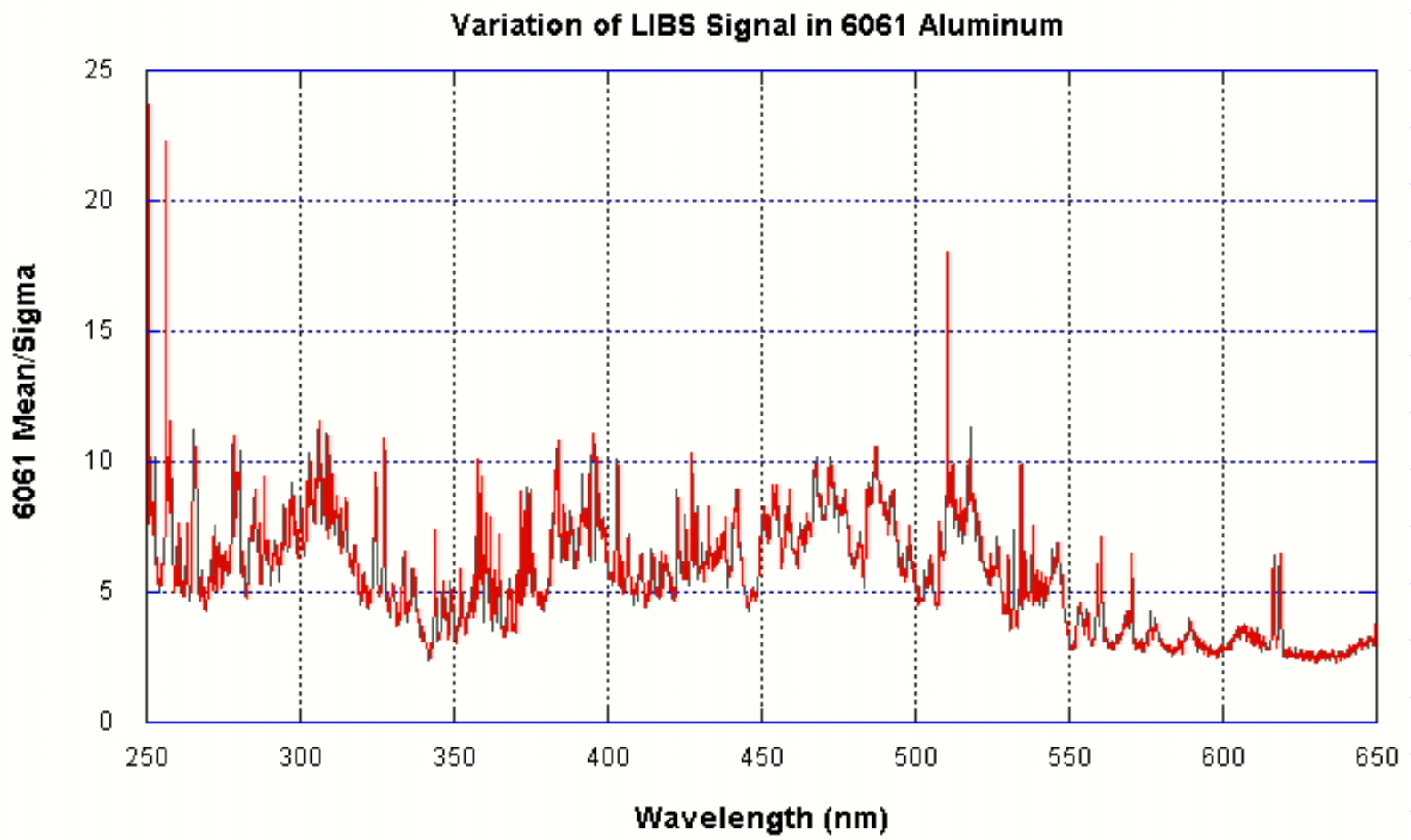

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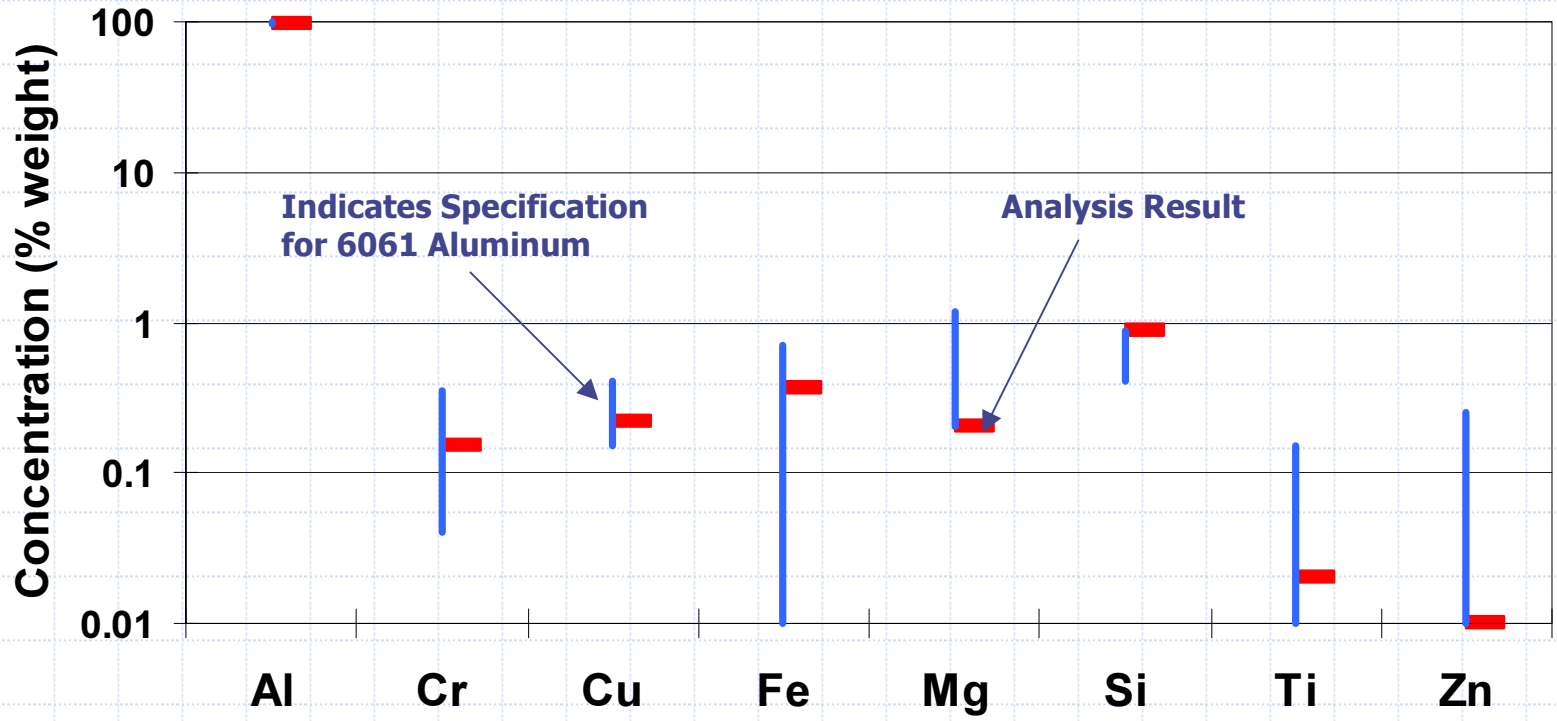

Total Potential Energy Savings

- Aluminum – 17 Trillion Btu per Year
- Glass – 17 to 45 Trillion Btu per Year
- Steel – 26 Trillion Btu per Year
- Total – 60 to 88 Trillion Btu per Year Possible

LIBS Advantages

- Fast
  - 10-50 Measurements/second with commercial low cost (~$10k) solid state lasers
- Accurate
  - Able to measure concentrations at fractions of a percent
- Applicable to a wide range of materials
  - Metals
  - Glass
- Proven
  - Extensive literature on use of the process in lab environments

Advantages

- Constituents Instantaneously and Accurately Determined Anywhere in the Melt
  - Replaces melt analysis based on single "button"
  - Can Drive Development of Computer Simulations of Furnace Performance by Providing Test Data
- Electronics Removed from the Harsh Melt Environment Through Use of Fiber Optics
- Overall System is Inexpensive

Conclusions

- LIBS Feasibility Proven
- Technology Tested in Pilot-Scale Facility
- Development Proceeding with Industry Guidance
- Multiple Number of Markets Identified – Some Not Originally Anticipated